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PRECISION DIFFRACTOMETRIC MEASUREMENTS OF THE ELEMENTARY CELL PARAMETERS*

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Precision measurements require the accounting and minimization whenever possible of all errors, random as well as systematic. Diffractometric investigation of polycrystalline materials is usually performed using the focusing arrangement of Bragg and Brentano for the X-ray beam diffracted by the horizontal plane. There is a number of reasons for the displacement of the diffracted beam from the correct Wolf-Bragg position. The sources of systematic errors, specifically in diffractometric investigations, are: (1) deviation of the plane of the sample being investigated from the exact focusing surface, which leads to the necessity of taking into consideration the influence of the "horizontal" deviation of the primary beam; (2) penetration of X-rays inside the sample; (3) shift of the reflecting plane of the sample from the goniometer axis; (4) vertical divergence of the reflected from

the primary beam; (5) incorrect setting of the counter zero. Apart from these sources of error related to the geometry of plotting in diffractometric investigations with continuous recording of the diffraction curve, there occur additional systematic errors related to the performance of the electronic integration circuit and to the inertia of the recorder. The influence of various systematic errors on the precision of diffractometric determinations of parameters of an elementary cell, as well as the correction for their influence by extrapolation to $\theta = 90°$, have been investigated many times /1,2,3, 4,5,11/. There is no need to take into consideration the refraction of X-rays in diffraction measurements; this should be done only where extremely precise measurements are desired.

Account of systematic errors

The determination of the reflection angles can be performed according to the position either of the maximum of the diffraction peak or of its "center of gravity". More precise results can be obtained from the determination of the position of the center of gravity. This procedure allows a more rigorous accounting of systematic errors and possesses a number of other advantages: (a) the determinations are based on the use of intensity measurements along the whole profile of the diffraction peak, and hence are more precise; the statistical error and the influence of random fluctuations have been substantially lessened; (b) the measurements do not depend

on the shape of the peak, the asymmetric deformation or the broadening of the peak; (c) the deviation of the center of gravity from the Wolf-Bragg position due to geometric deformations can be calculated more precisely than the corresponding deviations of the peak maximum. Let us give the expressions for the shift of the center of gravity for different geometric deformations.

The deviation of the sample plane being investigated from the focusing surface leads to a shift in the center of gravity depending on ctg θ. Since $\Delta d/d = - \mathrm{ctg}\theta \, \Delta\theta$, the error $\Delta d/d$ is proportional to $\mathrm{ctg}^2\theta$ or to $\cos^2\theta$ with accuracy to members containing $\cos^4\theta$.

The finite thickness of the reflecting layer, determined by the magnitude of the absorption coefficient, causes a shift in the position of the center of gravity by the amount $\sin 2\theta/4\mu R$. Hence, $\Delta d/d \sim \sin 2\theta . \mathrm{ctg}\theta = \cos^2\theta$.

Deviation of the sample plane from the goniometer axis by an amount S causes an error in the determination of the position of the center of gravity $S.\cos\theta/R$, and consequently $\Delta d/d \sim \cos\theta . \mathrm{ctg}\theta$ or to $\cos^2\theta$ (with accuracy to members of a higher order of smallness).

The vertical divergence of the primary from the reflected beam causes a shift in the position of the center of gravity

$$\Delta\theta^b = G_1 \, \mathrm{ctg}\, 2\theta + G_2 \cos\theta \, 2\theta$$

where $G_1 = \frac{1}{12R^2}\left(H_f^2 + H_c^2 + H_p^2\right)$; $G_2 = \frac{1}{6R^2} H_p^2$ (1)

H_f, H_c, H_p are the height of the focus, the height of the target before the counter, and the height of the illuminated part of the sample, respectively. The error

$$\Delta d/d = -\mathrm{ctg}\theta\ \Delta\theta = -Q_1 + \frac{Q_1 + Q_2}{2\sin^2\theta}$$

or with accuracy up to members containing $\cos^4\theta$.

$$\frac{\Delta d}{d} = -\frac{Q_1 - Q_2}{2} + \frac{Q_1 + Q_2}{2}\cos^2\theta \qquad (2)$$

The influence of errors caused by vertical divergence can not be eliminated by extrapolation to $\theta = 90°$. After extrapolation there remains the term /5/

$$-a = -\frac{Q_1 - Q_2}{2} = -\frac{1}{24R^2}\left(H_f^2 + H_c^2\right) \qquad (3)$$

To decrease this remaining member, it is necessary to decrease the vertical divergence of the primary, as well as that of the reflected beams. To achieve this, Soller slits are placed between the focus and the sample, and also between the sample and the counter /5/. The use of slits leads to a substantial decrease in intensity; however, with a divergence of 2° between the beams, it allows the reduction of the remaining member to a magnitude $\Delta d/d = 4.10^{-5}$.

The uncertainty in the zero position of the counter causes a certain contant error $\Delta\theta$ independent of the angle of

reflection. The corresponding error $\Delta d/d$ is proportional to $\mathrm{ctg}\theta$. A very careful adjustment of the goniometer allows the reduction of the error in the setting of the counter zero, to a rather small value /6,7/.

For large angles θ, where the diffraction peak is broad, the angular factor $Q(\theta)$ (Lorenz and plarization), changes markedly within the confines of the peak, alters its profile and shifts the center of gravity. The shift of the center of gravity can be determined, by calculating its magnitude for the diffraction function $J(\theta)$ and for the product $Q(\theta).J(\theta)$.

$$\Delta\theta^{Q} = \frac{\int \theta\, Q(\theta) J(\theta)\, d\theta}{\int Q(\theta) J(\theta)\, d\theta} \quad \text{where} \quad \theta_0 = \frac{\int \theta\, J(\theta)\, d\theta}{\int J(\theta)\, d\theta} \qquad (4)$$

The integration is performed within infinite limits. [Translator's Note: Probably what is meant is "from -to + ∞]

Expanding $Q(\theta)$ as a Taylor series in the neighborhood of θ_0 and taking the first two members, we will obtain:

$$\Delta\theta^{Q} = \frac{1}{Q}\left.\frac{\partial Q}{\partial \theta}\right|_{\theta=\theta_0} \cdot \frac{\int \delta^2 J^*(\delta)\, d\delta}{\int J^*(\delta)\, d\delta} \qquad (5)$$

where $\delta = \theta - \theta_0$ and $J(\theta) = J^*(\theta - \theta_0)$.

Substituting the expression for the angular factor in the powder method $Q(\theta) = \sin^2\theta\cos\theta/(1 + \cos^2 2\theta)$, and approximating the profile of the diffraction peak by the Gaussian function $\frac{1}{\sqrt{2H}\,\sigma} e^{-(\delta^2/2\sigma^2)}$ we will obtain

$$\Delta\theta^q = \left[tg\,\theta_0 + \frac{3\,ctg\,2\theta_0}{1+\cos^2 2\theta_0} \right] \sigma^2 \qquad (6)$$

For large angles θ the angular dispersion changes within the confines of the diffraction peak and this leads to the fact that the center of gravity of the diffraction peak (θ_0) does not coincide with the magnitude $\theta_c = \text{arc sin}\,\frac{\lambda_c}{2d}$ [λ_c is the center of gravity of the profile $\nu(\lambda)$ of the spectral line]. The correction which must be applied for the transformation from θ_0 to θ_c is

$$\Delta\theta^p = -\left[\frac{\int \theta J(\theta)\,d\theta}{\int J(\theta)\,d\theta} - \text{arc sin}\,\frac{\lambda_c}{2d} \right] \qquad (7)$$

Changing the first member to the new variable and taking into consideration that $J(\theta)d\theta = \nu(\lambda)d\lambda$, we will obtain

$$\Delta\theta^p = -\left[\frac{\int \text{arc sin}\,\frac{\lambda}{2d}\,\nu(\lambda)\,d\lambda}{\int \nu(\lambda)\,d\lambda} - \text{arc sin}\,\frac{\lambda_c}{2d} \right] . \qquad (8)$$

Expanding arc sin $\frac{\lambda}{2d}$ into a Taylor series around λ_c and taking only the first three members, we will have:

$$\Delta\theta'' = -\left[\frac{\partial}{\partial\lambda}\arcsin\frac{\lambda}{2d}\right|_{\lambda=\lambda_c}\cdot\frac{\int\delta\nu^*(\delta)\,d\delta}{\int\nu^*(\delta)\,d\delta}+$$

$$+\frac{\partial^2}{\partial\lambda^2}\arcsin\frac{\lambda}{2d}\left|_{\lambda=\lambda_c}\cdot\frac{\int\delta^2\nu^*(\delta)\,d\delta}{\int\nu^*(\delta)\,d\delta}\right. \qquad (9)$$

where $\delta = \lambda - \lambda_c$.

If $\nu^*(\delta)$ is represented approximately as a Gaussian function, we will obtain finally:

$$\Delta\theta'' = -\tfrac{1}{2}\,tg^3\theta_c\left(\frac{\sigma}{\lambda}\right)^2 \qquad (10)$$

The last two errors are not eliminated by the extrapolation, and corrections for them, in case of necessity, should be applied to each line separately. However, these corrections are usually not considerable.

Thus, careful preparation and adjustment of the apparatus allow (using the extrapolation of the parameters obtained, to $\theta = 90°$) the elimination of a number of systematic errors caused by the geometry of the plotting, apart from the vertical divergence. The curve of $\cos^2\theta$ used in the extrapolation is nearly a straight line. In order to improve the resulting precision, it is necessary to decrease the vertical divergence

to a value at which the magnitude of the remaining member would be less than the limit of precision. As has already been pointed out above, the use of Soller slits allows reduction of the vertical divergence, but leads to a substantial decrease in intensity. This decrease in intensity is particularly large in the case of a relatively small projection height of the focused spot. Another possible way of decreasing the vertical divergence is to place a focusing monochromator between the tube and the goniometer (Fig. 1). In this case the geometry of the plotting in the equatorial plane of the goniometer is the same as usual, only the focus of the curved monochromator is substituted for the focus of the tube; however, the use of a focusing monochromator permits a substantial increase in the range of reflection angles recorded. The path of the rays within the vertical plane changes, decreasing the vertical divergence. The crystal of the monochromator, placed at a certain distance f from the focus of the tube, becomes almost like a slit limiting the vertical divergence of the primary beam; the distance between the focus of the tube and the sample is increased. The beam of rays hitting the sample has a variable intensity along the height Z_p of the sample; photometric evaluation of a photographic plate placed instead of a sample showed that the intensity of the monochromatized beam hitting the sample can be represented by a trapezoid function of Z_p, related to the presence of half-shadows (Fig. 2).

Considering the abovementioned, we will obtain for the magnitude of the shift /$\Delta\theta^b$/ arising as a consequence of vertical divergence:

$$\Delta\theta^b = \left(\mathcal{X}_1 \frac{1-q}{1+g} + \mathcal{X}_2 + \mathcal{X}_3 + \mathcal{X}_y \frac{1-q}{1-g}\right) ctg\,\theta + \mathcal{L} \frac{1-q}{1-g} \cos 2\theta \quad (11)$$

where

$$\mathcal{X}_1 = \frac{H_p^2}{48(R+2f)^2} \; ; \quad \mathcal{X}_2 = \frac{H_f^2}{48(R+2f)^2} \; ; \quad \mathcal{X}_3 = \frac{H_c^2}{48R^2}$$

$$\mathcal{X}_y = \frac{H_p^2}{48R^2} \; ; \quad \mathcal{L} = \frac{H_p^2}{24R(R+2f)} \; ;$$

$$q = \frac{1}{H_p^3} 2K\left[\left(\frac{H_p}{2}\right)^3 \left(\frac{3}{2}H_p - h_p\right) + h_p^4\right]$$

$$g = \frac{1}{H_c} K\left[H_p\left(\frac{H_p}{4} - h_p\right) + h_p^2\right]$$

and K, h_p are according to Fig. 2.

The remaining member in the extrapolation formula is:

$$a = \frac{1}{2}\left[\frac{H_p^2}{48(R+2f)^2}\frac{1-q}{1-g} + \frac{H_f^2}{48(R+2f)^2} + \right.$$

$$\left. + \frac{H_c^2}{48R^2} + \frac{H_p^2}{48R^2}\frac{1-q}{1-g} - \frac{H_p^2}{24R(R+2f)}\frac{1-q}{1-g}\right] \quad (12)$$

Choice of the experimental condition R ≈ 2f will lead to mutual cancellation of the last two terms. Using the GUR-3

and a focusing monochromator of the type used in KMSP /8/, it is possible to obtain $a = 4.10^{-5}$ at R = 160mm, f = 75mm, H_f = 2.5mm, H_p = 14mm, h_p = 4mm and H_c = 8mm. The error can be lowered to 1.10^{-5} by decreasing the height of the illuminated portion of the sample to H_p = 6mm and of the height of the counter slit H_c to 4mm; however, at the same time the intensity decreases fourfold.

The adaptation of GUR-3 for precision measurements of elementary cell parameters.

It is well known that the precision with which the parameters of a cell can be determined increases with increasing reflection angle. The most important angle interval for precision measurements is $\theta = 75 \div 85°$. Measurements at angles $\theta = 85 \div 90°$ are hampered by a number of experimental difficulties and hindered by the influence of the natural spectral line-width of the characteristic radiation.

The limiting angle 2θ in GUR-3 that can be recorded is 150°. This is due to the construction of the stand carrying the counter and of the holder of slits defining the primary beam. The holder for flat samples in the GUR-3 is not built for regulation of the sample plane position. To adapt the goniometer GUR-3 for precision measurements, a focusing monochromator with an elastically bent crystal (Fig. 1) was substituted for the slit-holder. A quartz crystal with its large rhombohedron plane $10\bar{1}1$ serving as the reflecting surface was

placed in a crystal-holder KMSP /8/. Due to the small size of the crystal-holder and to the fact that it was placed at a distance R + f = 235mm from the goniometer axis, the counter could rotate to an angle 2θ = 168°. The stand supporting the monochromator was constructed in such a way as to allow precise adjustment of the monochromator, shielding from scattered rays, and the selection of a sufficiently narrow part of the spectrum. The sample-holder was modified so as to allow the positioning of the sample plane, with respect to the rotation axis of GUR-3, to be possible with a precision of a few microns. In positioning the sample plane, motion along the guide-rail is achieved through the use of a differential screw (the thread pitch of the screw in the moving part differed by 0.1mm from that of the screw in the stationary part).

One of the constructed modifications of the sample-holder could rotate around the normal to the plane being investigated, at a rate of 20 revolutions per minute. A place was provided for a special thermostat which could keep the temperature of the sample constant to ± 0.1°. The method of stabilizing the temperature is analogous to the one used in the RKVT /9/.

Precision measurements require very careful installation and adjustment of the apparatus. Apart from the usual adjustment of the goniometer, it is necessary to do the following: (a) the position of the monochromator must be adjusted so that the reflecting plane of the crystal is placed at an angle

corresponding to the chosen wavelength, and that the crystal itself is evenly illuminated. The unused radiation (of other wavelengths) must be kept out with the help of baffles. The focus of the monochromator is a line about 7mm long and less than 0.2mm wide, parallel to the goniometer axis and located on the circle of focusing. For the GUR-3, the monochromator focus must be located 160mm from the goniometer axis. The vertical plane passing through the monochromator axis and the main axis of the goniometer is the zero plane, starting from which the angles Θ must be counted. The reflected monochromatic beam must be bisected by the equatorial plane of the goniometer passing through the mid-points of the monochromator focus, the sample and the counter slit.

(b) The zero positions of the counter and of the flat sample-holder must be determined. In the zero position the average vertical slit axis and the sample plane to be investigated must coincide with the zero plane of the goniometer. Also, the positions of the goniometer and of the monochromator must be fixed so that the adjustment of the apparatus would not change during the experiment.

The zero settings of the counter and of the sample-holder, and also the coincidence between the sample plane investigated and the goniometer axis, must be checked periodically and definitely every time after changing the orientation of the monochromator from one wavelength to another. To determine and

set the zero positions of the counter and the sample plane, it is convenient to use a wedge and a special portable slit (Fig. 3). By placing the portable slit in place of the sample and another narrow slit 0.25 or 0.1mm wide, near the counter, and observing the position of the counter giving the maximum rate of counting for two positions of the portable slit differing from each other by 180° (W_0 and W_{180}), it is easy to determine the zero positions with a precision of a few minutes /6/. Using the wedge one can determine the zero positions with greater accuracy. The method consists in the following: the wedge is used to shut off first one, then the other half of the primary beam area falling on the counter /7/. The intensity registered by the counter will remain constant for positions of the wedge differing by 180°, only in the case where the counter slit is symmetrical relative to the zero plane. If the working plane of the wedge and hence the sample plane lies in the zero plane, the intensity will be equal to half the intensity registered with the wedge in the remote position. With the counter in the correct position, the intensity registered is equal to half the sum of intensities $\left[(J_0 + J_{180})/2\right]$ registered by the counter with its slit in an incorrect position. A sharp unevenness in the intensity of the primary beam can lead to an error in the determination of the zero positions by the wedge method. In cases where the goniometer permits measurements within rather extensive regions of positive as well as negative reflection angles, the zero position of the counter (γ_0) can be determined

by measuring the angles 2θ in the negative and the positive regions. In this case $\gamma_0 = (2\theta_+ + 2\theta_-)/2$. The determinations of the zero position performed by the above two methods led to the results which agreed with each other within the experimental error, 0.1 - 0.2 minutes.

Determination of the position of the center of gravity of diffraction peaks.

The position of the center of gravity of the diffraction peak is determined in the following manner

$$2\theta_c = \frac{\int_{-\infty}^{\infty} J(2\theta)\, 2\theta\, d(2\theta)}{\int_{-\infty}^{\infty} J(2\theta)\, d(2\theta)} \cong \frac{\sum_{K=1}^{n} J_K 2\theta_K}{\sum_{K=1}^{n} J_K} \qquad (13)$$

where J_k is the intensity averaged over the width of the slit with the middle of the counter slit at $2\theta_k$.

Determination of the center of gravity of a diffraction peak is long and tedious. In attempts to find a short cut we investigated the dependence of the error in the determination of the center of gravity, on the precision of the intensity measurements at different points along the peak profile, on the number of points, on the counter slit width, and on the magnitude of the intervals between measurements. In the presence of a background the center of gravity was determined in the following way: the intensity (J_k) was measured at the "K" points of the

profile separated by a constant interval (h = 5'), and the intensity of the background was measured at two points sufficiently removed from the peak maximum (Fig. 4). The center of gravity can then be obtained from the following expression:

$$2\theta_c^m = \frac{M_m}{S_m} = \frac{M_J - M_\phi}{S_J - S_\phi} \qquad (14)$$

where M_ϕ is the moment of the area of the background taken as linear, S_ϕ is the area of the background, while $M_J = \Sigma J_k 2\theta_k$ and $S_J = \Sigma_J = \Sigma J_k$.

Assuming that the errors J_k and J_ϕ of the measurements are independent, that the peak is of Gaussian shape and neglecting higher order terms, we will obtain the following expression for the error in the position of the center of gravity:

$$\Delta 2\theta_c^m = \left\{\left[\varepsilon\left(\frac{J_{max}}{2}\right)\frac{nh}{\sqrt{n}}\right]^2 + \left[\frac{S_\phi}{S_m}\varepsilon(J_\phi)\left(\frac{nh}{6} + 2\theta_{1/2} - 2\theta_c^m\right)\right]^2\right\} \qquad (15)$$

where $\varepsilon\left(\frac{J_{max}}{2}\right)$ and $\varepsilon(J_\phi)$ are relative errors in intensity measurements; n is the number of points measured.

To make the correct choice of the method of measurement we will consider the following:

(a) The dependence of the time of measurement at each point, on the distance to the center of gravity of the peak

(assuming that the error is mainly determined by the statistical fluctuations in intensity). For a peak of Gaussian shape and for different background values, the above functions are given in Fig. 5. It is seen from this figure that if the background is less than 1/4 of the maximum peak intensity, then it is rational to use the method of measuring intensities at constant counting time. The required counting time interval must be determined at the half-intensity point from the given magnitude of error.

(b) The dependence of the magnitude of the relative error on the distance from the center of gravity of the peak. For different background levels it is given in Fig. 6. It follows from these curves that for background levels higher than 1/4 of the maximum peak intensity, one must measure the intensities according to the method of constant number of impulses.

Adding the constant counting rate it is possible to obtain intermediary variations between the method of constant counting time and the method of constant number of impulses. The trick of adding the constant counting rate is most appropriate for measurements of the center of gravity of peaks. Choice of the magnitude of the added rate can be done from the curves of Fig. 7. Correct choice of the method of measurement of the intensity shortens considerably the time necessary for the measurements.

The measurements can also be accelerated by rejecting that part of the peak where the intensity is small. Calculations

for a Gaussian peak show that the error in the determination of the coordinates of the center of gravity due to the rejection of one wing of the peak where the intensity is less than 2% of the maximum is determined by the following expression:

$$\Delta 2\theta_c^m = 2\,\frac{\lambda}{1200} \qquad (16)$$

where λ is the peak half-width. Thus, measurements in regions where the intensity is less than 1% of the maximum are not justified.

Increasing the width of the counter slit increases the intensity and hence the rate of counting, but in general increases the shift in the position of the center of gravity of the peak.

The shift in the position of the center of gravity is determined by the fact that the counter slit cuts out of the area defined by the peak profile a curvilinear trapeze, whose center of gravity ($2\theta_c^k$) does not coincide with the position of the mid-point of the counter slit ($2\theta_k$) (see Fig. 5). The change in the statistical moment of the trapeze relative to the moment of the rectangle is

$$\Delta M_k = \Delta 2\theta_c^k S_k = \int \mu\, J(2\theta_k + \mu)\, d\mu \qquad (17)$$

where S_k is the area of the trapeze.

Limiting ourselves to the first member of the expansion into series and hence to a precision determined by the higher-order members, we will obtain

$$\Delta 2\theta_c^m = \frac{1}{S_m}\left\{\frac{b_c^2}{12}\left[J(b) - J(a)\right] + \frac{b_c^3}{12}\left[J'(b) - J'(a)\right]\right\} \quad (18)$$

where S_m is the peak area, "a" and "b" are the initial and the final points, J'(a) and J'(b) are the derivatives $\frac{dJ(2\theta)}{d(2\theta)}$ at points "a" and "b" correspondingly, and b_c is the angular counter slit-width. Assuming J(b) = J(a) we will obtain

$$\Delta 2\theta_c^m = \frac{b_c^3}{12 S_m}\left[J'(b) - J'(a)\right] \quad (19)$$

The use of a comparatively wide slit (1mm = 20') causes a sufficiently small (<0.05') shift in the position of the center of gravity, but at the same time increases the general intensity measured at each given slit position, and hence makes the measurements of the center of gravity more reliable and less time-consuming.

<u>Measurements of the parameters of the cell in tungsten from determinations of the centers of gravity of the lines</u>.

Tungsten powder with a small amount of cellulose nitrate lacquer added, was packed into the hole (15mm in diameter and 0.5mm deep) in a plate of "organic glass". [Translator's

Note: "Organic glass" is a term frequently used to describe a group of synthetic polymers, e.g., methyl methacrylate, allyl esters of polybasic acids.] The surface of the sample was leveled with a glass plate. Since the average dimensions of the crystallites were less than 5μ the X-ray diffraction measurements were performed without rotating the sample. The intensities were measured at each point. The time for each point was 1 min; for very weak lines in the peripheral regions, 4 min. The background was taken as the average of five measurements or at one point for a duration of 10 min. The counter slit-width was 1mm, its height 8mm. The angular shifts during peak measurements were 5'. The temperature was controlled by a thermometer placed near the sample. The temperature deviation during measurement of a peak did not exceed 0.5°C. The results of the measurements were reduced to 25°C. In order to eliminate the influence of the doublet the measurements were performed with the Cu K_β radiation (λ_c = 1.39223 Å *). The average error in the measurement of the position of the center of gravity of the peak was ± 0.3'. Repeated measurements performed with different modifications of the goniometer and the monochromator showed that the error caused by the nonreproducibility of the

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*$\lambda_c = \lambda_{max} + \Delta\lambda$; $\Delta\lambda$ is not zero if the spectral line is asymmetric. Approximating the line profile with a triangle having the same half-width (a) and coefficient of asymmetry (α) we will obtain $\Delta\lambda = \frac{2}{3} a \frac{\alpha-1}{\alpha+1}$. For the line CuK_β a = 0.0010 Å, α = 1.18 /10/ whence $\Delta\lambda \approx 0.00006$ Å and $\lambda_c = 1.39217 + 0.00006 = 1.39223$ Å.

installation (of the apparatus) surpasses (by 2-2.5 times) the error in the measurement of the position of the center of gravity of a peak. Thus relative measurements of the positions of the center of gravity of a peak, and hence the relative measurements of parameters can be performed with a higher degree of accuracy than absolute measurements.

Corrections for dispersion and angular factor at $\theta = 80°$ evaluated from equations (6) and (10), correspond to $\Delta\theta^D < 0.03'$ and $\Delta\theta^Q < 0.25'$ correspondingly. Both corrections are negative. Due to their smallness we neglected to apply these corrections. The results of measurements on three lines (321, 411-330 and 420) are given in the table.**

Hence a = 3.16526 ± 0.00005 Å.

Table No.

Parameter a of tungsten determined with the Cu K_β radiation.

hkl	$2\theta_c^m$	abÅ	$\cos^2\theta$
321	110° 44.4'	3.16540	0.323
411 and 330	137° 10.5'	3.16513	0.130
420	149° 11.5'	3.16504	0.033

** Plottings performed with the apparatus using another goniometer gave values for the angles which deviated systematically from the results given above. This indicates the necessity for a supplementary precise angular gradation of the goniometer in order to obtain precision results.

Performing the extrapolation according to $\cos^2\theta$ we obtain a = 3.16500 Å. The correction for refraction was introduced according to the expression:

$$(1-n)a = 4.47 \cdot 10^{-6} \left(\frac{A}{a}\right)^3 \Sigma A \qquad (20)$$

where n is the index of refraction; ΣA is the sum of the series numbers of all atoms constituting the cell. For tungsten and using the Cu K_β radiation the correction for refraction is 12.8×10^{-5} Å.

The correction for the vertical divergence, determined from equation (12), is 13×10^{-5} Å.

Hence a = 3.16526 ± 0.00005 Å.

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Fig. 1. Apparatus for precision plotting using a URS-501. T - tube; F - focus of the tube; g - goniometer; M - monochromator; D - sample holder; C - counter; P - sample; S_1 and S_2 - monochromator slits; S_3 - counter slit.

Fig. 2. Vertical divergence of the primary beam in using the monochromator. The curve for the illumination of the sample I(2p) is obtained through microphotometric evaluation.

Fig. 3. Device for adjustment.
(a) Brass wedge for determination of counter zero.
(b) Slit to determine the zero position of the sample plane.

Fig. 4. For the determination of the center of gravity of a diffraction peak.

Fig. 5. Dependence of the time of measurement on the distance from the center of gravity of the line. $\Delta(J_k S_k)$ = const. The ratio of the intensities $J_\phi/(J_{max}-J_\phi)$ is as follows: 1-0; 2-1/120; 3-1/8; 4-1/4; 5-2/5; 6-1/2; 7-5/6; 8-3/4; 9-7/8; 10-1.
The profile of the diffraction peak is shown by the dotted line.

Fig. 6. Dependence of the magnitude of the relative error in intensity measurements, on the distance to the center of gravity. $\Delta(J_k S_k)$ = const.
The labeling of the curves is the same as in Fig. 5.

Fig. 7. Dependence of the magnitude of the relative error in intensity measurements on the magnitude of the intensity. $\Delta(J_k S_k)$ = const.
The labeling of the curves is the same as in Fig. 5.